[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO 16.]

THE SEPARATION OF VANADIUM FROM ARSENIC.

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Received October 2, 1896.

AS vanadium and arsenic occur associated in minerals and likewise in artificial products, their separation becomes a matter of consequence.

The course usually pursued in carrying out this separation is that long since recommended for the removal of vanadic acid from its solutions; namely, its precipitation as ammonium metavanadate. Other methods have recently appeared in the literature bearing on analysis. Reference is here made especially to the publication of Fischer.[1]

Experiments made in this laboratory on the behavior of vanadates[2] and arsenates[3] heated in an atmosphere of hydrochloric acid gas, in which both acids were volatilized, suggested the thought that if the sulphides of vanadium and arsenic were exposed to the same vapors perhaps they would show a variation in deportment. And so it has proved. Perfectly dry arsenic trisulphide, previously washed with alcohol, carbon disulphide, and ether, then dried at 100° C., when exposed in a porcelain boat, placed in a combustion tube, was almost completely expelled from the retaining vessel at the ordinary temperature. The last traces were driven out at a temperature little above 150° C. Brown vanadium sulphide, in a perfectly dry condition, treated in the same manner, was not altered. It only remained then to prepare mixtures of known amounts of the two sulphides and subject them to the action of the acid vapor. To this end the following experiments were made:

I. 0.1303 gram of vanadium sulphide,
0.1302 gram of arsenic sulphide.

The arsenic sulphide was volatilized without difficulty and left 0.1297 gram of vanadium sulphide.

[1] Bestimmung von Vanadinsäure: Dissertation, Rostock, 1894.
[2] *J. Am. Chem. Soc.*, 16, 578.
[3] *Ibid.*, 17, 682.

II. 0.1290 gram of vanadium sulphide,
0.2242 gram of arsenic sulphide,

gave after exposure of one hour to hydrochloric acid vapor a residue of vanadium sulphide, weighing 0.1297 gram.

III. 0.0828 gram of vanadium sulphide.
0.0582 gram of arsenic sulphide,

left 0.0827 gram of vanadium sulphide.

IV. 0.1306 gram of vanadium sulphide,
0.2028 gram of arsenic sulphide,

gave a residue of 0.1308 gram of vanadium sulphide.

V. 0.1403 gram of vanadium sulphide,
0.2409 gram of arsenic sulphide,

left 0.1404 gram of vanadium sulphide.

The temperature in these experiments was not allowed to exceed 250° C., as beyond that point there is danger of affecting the vanadium and causing its partial volatilization.

The method worked so well and with such evidently favorable results that the following course was adopted in the analysis of a specimen of the mineral vanadinite. 0.2500 gram of air-dried and finely divided material was placed in a porcelain boat; the latter was then introduced into a combustion tube and gently heated in a current of dry hydrochloric acid gas. By this treatment vanadic and arsenic oxides were expelled, leaving lead phosphate and chloride. The receiver containing the vanadium and arsenic was made alkaline and digested with ammonium sulphide. From the solution of the sulpho-salts the vanadium and arsenic sulphides were set free by a dilute acid. After washing and careful drying these sulphides were separated as indicated in the preceding lines, then changed to oxides and determined in the usual manner. The sum of the total constituents determined as lead oxide, phosphoric oxide, vanadic and arsenic oxides, with some lead chloride, amounted to 0.2501 gram.

The method in addition to being satisfactory in the analytical way, certainly forms a very excellent means of purifying and freeing vanadium from arsenic.